Project Title
Hydrogen Production from Hydrogen Sulfide in IGCC Power Plants

Final Scientific /Technical Report

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ABSTRACT

IGCC power plants are the cleanest coal-based power generation facilities in the world. Technical improvements are needed to help make them cost competitive. Sulfur recovery is one procedure in which improvement is possible. This project has developed and demonstrated an electrochemical process that could provide such an improvement.

IGCC power plants now in operation extract the sulfur from the synthesis gas as hydrogen sulfide. In this project H_2S has been electrolyzed to yield sulfur and hydrogen (instead of sulfur and water as is the present practice). The value of the byproduct hydrogen makes this process more cost effective. The electrolysis has exploited some recent developments in solid state electrolytes.

The proof of principal for the project concept has been accomplished.

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1. Executive Summary

The capital cost for an IGCC power plant is greater than for a conventional plant. This is partly justified by higher efficiency, lower emissions and the potential for producing byproducts. However, the bottom line is that conventional coal and natural gas fired power plants can produce electric power at a lower net cost. Although increased fuel costs and environmental stipulations may eventually compensate for this difference, technical improvements are needed to help achieve this and remove the economic barrier to commercialization of these cleaner and more efficient systems.

The sulfur recovery systems of IGCC power plants can be improved and thereby produce an additional revenue stream that will lower the net cost of IGCC electricity. The current IGCC power plants in operation extract the sulfur from the synthesis gas as hydrogen sulfide. The hydrogen sulfide is extracted along with carbon dioxide in a stream called acid gas. Partial oxidation of the acid gas with air yields elemental sulfur and water (Claus Process) with a stream of dilute carbon dioxide in nitrogen[1]. The nitrogen would make sequestration of the carbon dioxide difficult. The energy cost of decomposing H_2S and the voltage required to do it electrolytically are several times lower than for decomposing water. Electrolysis of hydrogen sulfide yields sulfur and hydrogen (instead of sulfur and water). As a result, this is potentially such a cost effective procedure it can have a positive influence on the cost effectiveness of IGCC energy.

This project is developing better ionic transport membranes that can be used to produce hydrogen during the sulfur recovery processes in IGCC power plants at a low cost. These membranes can also be useful for separation of hydrogen from synthesis gas as well as for fuel cells. Fabrication of these membranes exploits some recent developments in solid state electrolytes[2]. These are inorganic crystals with which the hydrogen and sulfur in hydrogen sulfide can be cleanly separated with a low energy and voltage requirement. This report will describe the technique, apparatus and results of experiments done in decomposing hydrogen sulfide electrolytically.

In this project H_2S was decomposed electrolytically at 150C, a temperature at which sulfur is a low viscosity liquid so that it can run out of the electrolytic cell quickly and easily. This process has exploited some recent developments in solid state electrolytes. These involve inorganic crystals whose proton conductivities rise rapidly with temperature. One of these is cesium hydrogen sulfate which has a high conductivity at 150C[3].

Electrolytic cells were designed and fabricated for operating at 150C with gaseous H_2S and liquid sulfur in the anode compartment and hydrogen gas in the cathode compartment.

Apparatus for monitoring and control of the hydrogen sulfide, sulfur and hydrogen flows was designed and operated.

H_2S was decomposed electrolytically in experiments with a variety of electrode configurations and catalysts.

Sulfur produced was studied by XRD, DSC and SEM. Hydrogen gas produced was collected and measured by displacement of aqueous solution and identified by gas chromatography.

The primary objective of this work, which is to demonstrate a novel environmentally benign economical one-step pathway for dissociating H_2S electrolytically to produce hydrogen and liquid sulfur, has been attained.. In order to accomplish this task, we have: (1) investigated and developed a solid acid membrane, (2) fabricated a membrane exchange assembly (MEA) and electrolytic cell, and (3) implemented gas control and monitoring systems.

The proof of principal for the project concept has been accomplished.

2. REPORT DETAILS

2.1. INTRODUCTION

The capital cost for an IGCC power plant is greater than for a conventional plant. This is partly justified by higher efficiency, lower emissions and the potential for producing byproducts. However, the bottom line is that conventional coal and natural gas fired power plants can produce electric power at a lower net cost. Although increased fuel costs and environmental stipulations may eventually compensate for this difference, technical improvements are needed to help achieve this and remove the economic barrier to commercialization of these cleaner and more efficient systems.

All components should be examined for potential improvements that can improve IGCC cost effectiveness. The sulfur recovery system is one component of IGCC power plants that can be improved so as to produce hydrogen as a byproduct and thereby produce an additional revenue stream that will lower the net cost of IGCC electricity. The current IGCC power plants in operation extract the sulfur from the synthesis gas as hydrogen sulfide. The hydrogen sulfide is extracted along with carbon dioxide in a stream called acid gas. Partial oxidation of the acid gas with air yields elemental sulfur and water (Claus Process) with a stream of dilute carbon dioxide in nitrogen[4]. The nitrogen would make sequestration of the carbon dioxide difficult.

The energy cost of decomposing H_2S and the voltage required to do it electrolytically are several times lower than for decomposing water. Electrolysis of hydrogen sulfide yields sulfur and hydrogen (instead of sulfur and water). Analysis of a commercial size model has indicated that electrochemical dissociation of hydrogen sulfide waste streams in IGCC power plants can be economically lucrative. This is potentially such a cost effective procedure it can have a positive influence on the cost effectiveness of IGCC energy.

This project is developing better ionic transport membranes that can be used to produce hydrogen electrolytically during the sulfur recovery processes in IGCC power plants at a low cost. These membranes can also be useful for separation of hydrogen from synthesis gas as well as for fuel cells. Fabrication of these membranes exploits some recent developments in solid state electrolytes[5]. These are inorganic crystals with which the hydrogen and sulfur in hydrogen sulfide can be cleanly separated with a low energy and voltage requirement. This report will describe the technique, apparatus and results of experiments done in decomposing hydrogen sulfide electrolytically.

2.2. Approach

In this project H_2S was decomposed electrolytically at 150C, a temperature at which sulfur is a low viscosity liquid so that it can run out of the electrolytic cell quickly and easily. This process has exploited some recent developments in solid state electrolytes. These involve inorganic crystals whose proton conductivities rise rapidly with temperature. One of these is cesium hydrogen sulfate which has a high conductivity at 150C[6].

Electrolytic cells were designed and fabricated for operating at 150C with gaseous H_2S and liquid sulfur in the anode compartment and hydrogen gas in the cathode compartment.

Apparatus for monitoring and control of the hydrogen sulfide, sulfur and hydrogen flows was designed and operated.

H_2S was decomposed electrolytically in experiments with a variety of electrode configurations and catalysts.

Sulfur produced was studied by XRD, DSC and SEM. Hydrogen gas produced was collected and measured by displacement of aqueous solution and identified by gas chromatography.

H_2S introduced into the anode compartment of an electrolytic cell contacts the solid electrolyte and anode catalyst and is electrolytically decomposed to form liquid sulfur at the anode and gaseous hydrogen at the cathode at a temperature at which liquid sulfur has a low viscosity and can flow out of the electrolytic cell quickly and easily. The red curve in Figure 2.2 gives the viscosity of liquid sulfur and shows that it is minimized near 150°C[7]. We will operate near this temperature. To electrolyze H_2S we are seeking to exploit some recent developments in solid state electrolytes. These involve inorganic crystals whose proton conductivities rise rapidly with temperature. One of these electrolytes is cesium hydrogen sulfate whose conductivity is shown by the blue curve in Figure 2.2. The conductivity scale is logarithmic. The conductivity goes up by about 5 orders of magnitude between 120 and 150°C and the material is a good conductor at the temperature where we want to work[8].

The test-bed is an electrochemical cell in which a solid proton conducting membrane separates an anode chamber from a cathode chamber. The process consists of passing H_2S containing gas through the anode chamber to contact a catalytic anode, where it reacts to produce elemental sulfur, protons and electrons. The protons pass through the membrane from the anode chamber to the cathode chamber, where they react with electrons from the catalytic cathode to produce hydrogen gas. During the process, both the anode and the cathode are maintained at 150 ^{o}C, a temperature at which sulfur has a low viscosity and the solid electrolyte is in its hyperprotonic phase. Liquid sulfur is collected from the anode compartment and hydrogen is removed from the cathode compartment.

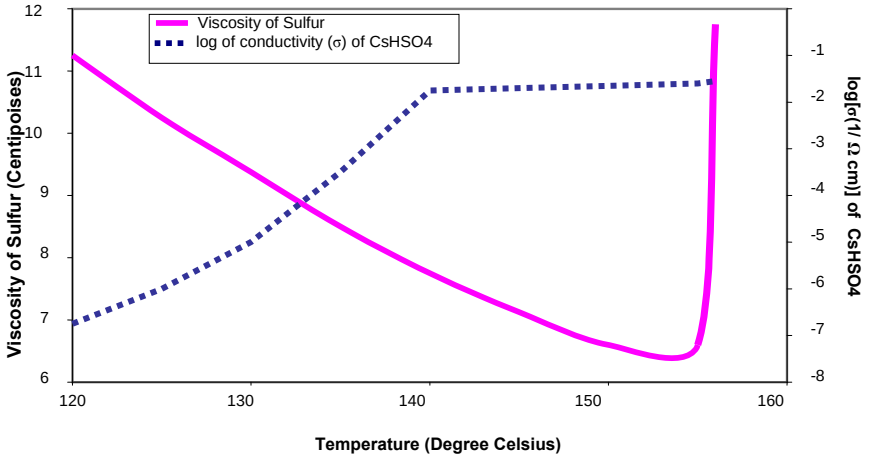

Fig 2. 2. Viscosity of Sulfur, Conductivity of CsHSO4 vs. Temperature[4]

2.3. EXPERIMENTAL METHODS

2.3.1. Electrolytic Cells

Two electrolytic cells, with a 2" and a ½" electrolyte respectively, are operational. They operate at 150C with gaseous H_2S and liquid sulfur in the anode compartment and hydrogen gas in the cathode compartment. The cells have thick walls to accommodate pipe fittings. The housing is chrome plated 316 stainless steel. Aflas o-rings and gaskets are employed to resist the corrosive anode contents.

Figure 2.3.1a is a photograph of the 2" cell. Figure 2.3.1b shows the cell mounted in an oven for operation at 150C.

Fig. 2.3.1a: Photograph of 2” electrolytic cell

Fig. 2.3.1b: Electrolytic cell in oven

A schematic of the 2” cell is shown in Figure 2.3.1c. This cell is similar to the 1/2 inch cell shown in Figure 2.3.1d but the ½ inch cell incorporates a sampling port with a septum through which hypodermic needles can be inserted to withdraw samples from the cell compartment.

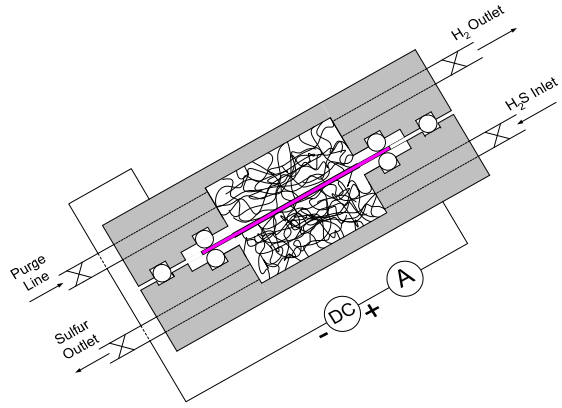

Fig.2.3.1c Schematic diagram of 2” test cell

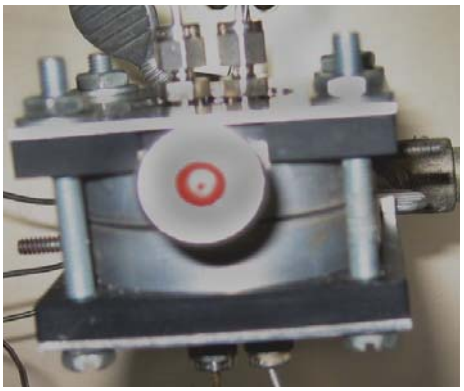

Fig. 2.3.1d. Photograph of ½” test cell

The sulfur compartments are modeled after the sulfur compartments in sodium sulfur batteries which are filled with carbon felt. This carbon felt, which is in direct contact with the electrolyte, conducts electrons from the electrolyte in the anode compartment. The same material is being used in the cathode compartment.

2.3.2. Flow Control

Apparatus for monitoring and control of the hydrogen sulfide sulfur and hydrogen flows has been designed and set up for operation. A liquid sulfur collection system has been set up and the water displacement apparatus for hydrogen produced is in place. A schematic of the system is shown in Figure 2.3.2.

Hydrogen sulfide is delivered to the anode compartment of the electrolytic cell via 316 stainless steel tubing regulated by a pressure regulator on the tank, monitored by a flow meter and controlled by a micrometering valve in the line. The pressure is continuously monitored.

The electrolytic cells use a 2 inch or ½ inch diameter solid electrolyte disc. Electrons are removed in the anode compartment to yield hydrogen ions and liquid sulfur. The liquid sulfur pools at the bottom of the compartment and runs out of a drain tube there for collection. The liquid pool seals the drain tube against outflow of hydrogen sulfide from the compartment. Hydrogen ions pass through the solid electrolyte to the cathode compartment where they gain electrons to form hydrogen gas that flows out at the top.

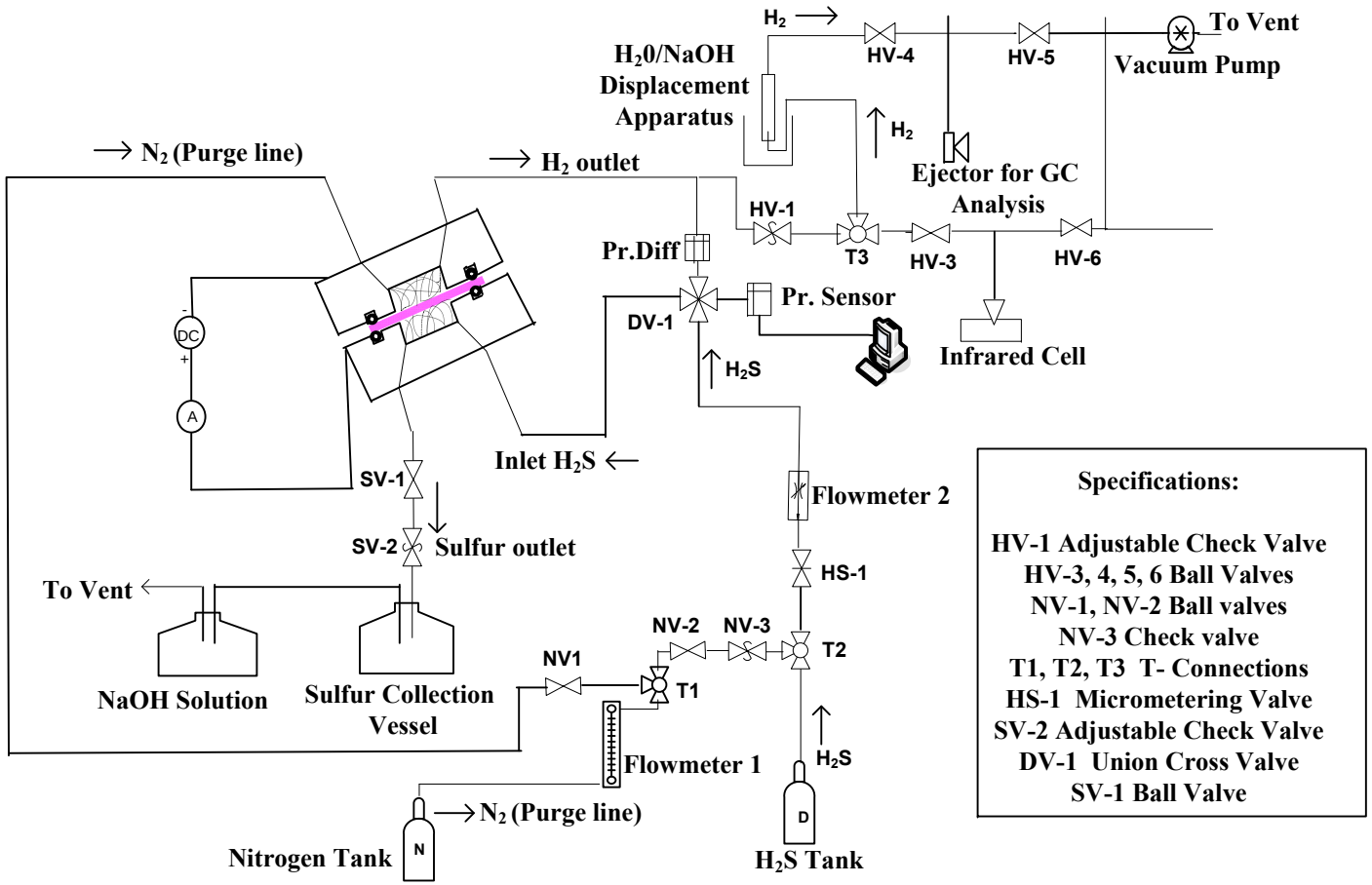

Fig.2.3.2: Schematic set-up of the electrolytic decomposition system

Two pressure transducers are used to monitor the pressures in the anode and cathode compartments for greatest sensitivity. Our intentions are to eventually make the electrolyte pellets as thin as possible to maximize their conductance[9]. This requires minimizing the pressure difference they must withstand. Hence this difference is carefully measured and kept small.

The hydrogen produced flows into a manifold from which samples are taken for various analyses. The gas is collected for analysis by displacement of water in a graduated container that immediately shows its volume. For safety, the water includes dissolved alkali to absorb any acid gas contaminants. The hydrogen so collected is analyzed by gas chromatography. The manifold directs some of the hydrogen to an infrared cell port with which the unpurified hydrogen can be analyzed for acid gases.

There are nitrogen purge lines for purging both compartments before and after the experiments.

2.3.3. Materials

Hydrogen sulfide information: Vendor: Specialty Gases of America, Purity = 99.5%, Mwt: 34.08, Specific volume: 1.23ft^3/lb, Labels: *P, *F: *P = Poison-inhalation hazard, *F = Flammable gas,ΔfH^0gas, -20.5kJ/mol (exothermic), Autoignition temperature, 500F or 260C, Density, 1.363g/L, gas slightly heavier than air, CGA-180-110 or (330 Airgas regulator can be used).

The body of the electrolyzer is made of chrome plated stainless steel.

The sources of starting materials and the purity of the catalysts and the electrolyte materials used were:RuO_2 (Sigma Aldrich, 99.9%), Pt black (Sigma Aldrich,99.9%), p-Dichlorobenzene (Sigma Aldrich 99.9%), $CsHSO_4$ (Sigma Aldrich, 99%), Sulfur (Sigma Aldrich, 99.98%).

2.3.4. Analytical Techniques

2.3.4.1. XRD Characterization

The powder X-ray diffraction of the sulfur product from electrolysis has been carried out by the Philips X'pert diffractometer with Cu Kα radiation of known wavelength. The incident and the diffraction slit width employed for the measurements are 1^o and 2^o respectively. Prior to this measurement, the diffraction from the paraffin tape was calibrated and the angles of occurrence were obtained. Phase identification and particle size calculations have been carried out by PANanalytical X'pert Highscore software version 1.0f[10].

2.3.4.2. Microstructure and chemical analysis

The microstructure of sulfur from the electrolysis product was obtained by Hitachi S800 scanning electron microscopy (SEM) and local composition was determined in the energy dispersive X-ray spectroscopy (EDS) mode using the same instrument. A fixed working distance of 10mm and a voltage of 25Kv were used. EDAX genesis software was used to analyze the SEM and EDS spectra.

2.3.4.3. Calorimetric measurement

Differential scanning calorimetry (DSC) was used to measure the heat flow of the reaction enthalpy in order to obtain the melting point of sulfur from the electrolysis product which was then compared to that obtained from pure sulfur. The ramp rate for this measurements was 2^{o} C/min. TA's Universal Analysis 2000 software program was used to analyze the DSC profiles.

2.3.4.4. Gas Chromatography

Hydrogen produced in the cathode compartment was identified by gas chromatography employing an Agilent 6890N gas chromatograph with a thermal conductivity detector.

2.3.5. Pellet Impermeability

Pellet permeability has been studied in the ½" cell. First both compartments were flushed with nitrogen. Then the compartment with the port was sealed off and the other compartment flushed with test gas. Samples were taken from the port periodically and the presence of test gas in the samples indicated whether the pellet is permeable to it.

2.4. RESULTS AND DISCUSSIONS

2.4.1. Electrolysis

Experiments have been conducted with electrodes pressed into the electrolyte and with the electrodes more loosely attached.

Hydrogen sulfide has been electrochemically decomposed with the ½" cell in an array of experiments involving different electrode attachments, catalysts and voltages. Results are given in Table 2.4.1 The annotations "anode+PC" and "cathode+PC" as used in Table 2.4.1 imply that carbon material is pressed into the electrolyte surface on the anodic side and the cathodic side of the pellet respectively. The annotation "Cfelt" means that the anode and cathode compartments are filled with carbon felt which aids in conducting the electrons to and from the surface of the electrolyte.

An experiment using anodic catalysts comprising of ruthenium (IV) oxide/cesium hydrogen sulfate/pt black/ p-Dichlorobenzene and cathodic catalyst comprising of platinum black/p-Dichlorlbenzene has given the best result so far and will be used as a benchmark for further development.

The total exposed surface on each side of the electrolyte pellet is 0.4 cm^2. The active area is less because of electrode coverage. The pellet thickness is 0.1 cm. The small area to thickness ratio implies a large ohmic resistance. Work on reduction of pellet thickness is under way.

Table 2.4.1: Effect of system configurations on the electrolysis process

Experiment	1	2	3	4	5	6	7	8	9	10	11	12
Electrode	Cfelt	Cfelt	Cfelt	Cfelt	Cfelt	Cfelt	Cfelt	Cfelt	Cfelt	Cfelt	Cfelt	Cfelt
Anode+PC	N	N	Y	Y	Y	Y	Y	Y	N	N	Y	N
Cathode+PC	N	Y	N	Y	Y	Y	Y	Y	Y	N	Y	Y
Anode cat.	N	N	N	N	N	RuO_2	RuO2	WC	RuO_2	N	V_2O_5	N
Cathode cat	N	N	N	N	Pt.b	Pt.b	N	Pt.b	Pt.b	Pt.b	Pt.b	Pt.b
Voltage(V)	m A	m A	m A	m A	m A	m A	m A	m A	m A	m A	m A	m A
0.9	0.21	0.37	0.38	0.30	0.50	1.90	0.36	0.29	0.40	0.33	0.36	0.30
1.2	0.26	0.37	0.65	0.51	0.70	3.80	2.40	0.38	3.10	0.33	0.49	0.37
1.4	0.26	0.37	0.7	0.51	1.60	5.10	2.70	0.38	3.90	0.35	0.62	1.20
1.6	0.27	0.37	0.78	1.00	3.20	6.10	2.80	0.39	5.70	0.36	0.70	1.75
1.8	0.28	0.38	0.95	2.00	3.40	7.00	2.90	0.40	7.80	0.43	0.70	1.75
2.0	0.28	0.40	0.95	2.10	3.40	7.50	2.90	0.54	7.80	0.49	0.70	1.75

Figure 2.4.1 shows current density as a function of voltage based on the total exposed surface for experiment 6 in Table 2.4.1. At lower currents the curve is linear as Ohms law. At higher currents diffusion limitations seem to be coming into play.

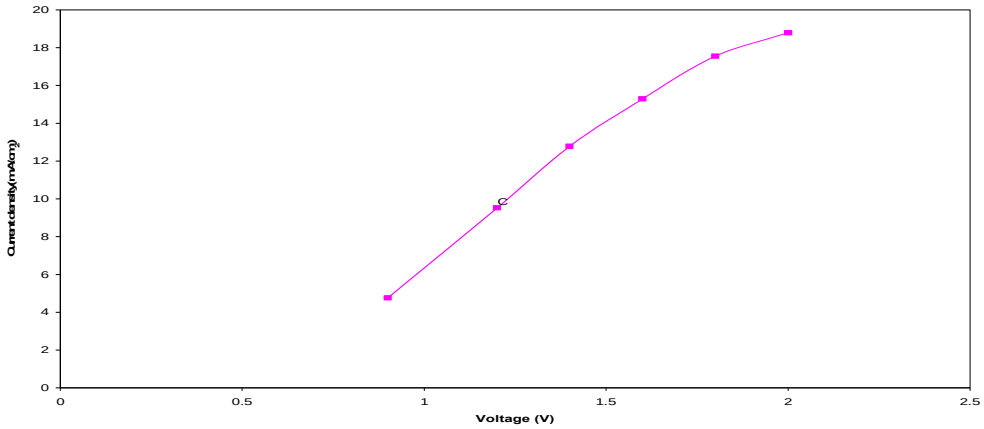

Fig 2.4.1: Current density vs. voltage generated from Table (1) for experiment (6).

2.4.2. Product Analysis

Sulfur produced in the anode compartment was characterized using X-ray diffraction (XRD) an scanning electron microscopy (SEM).

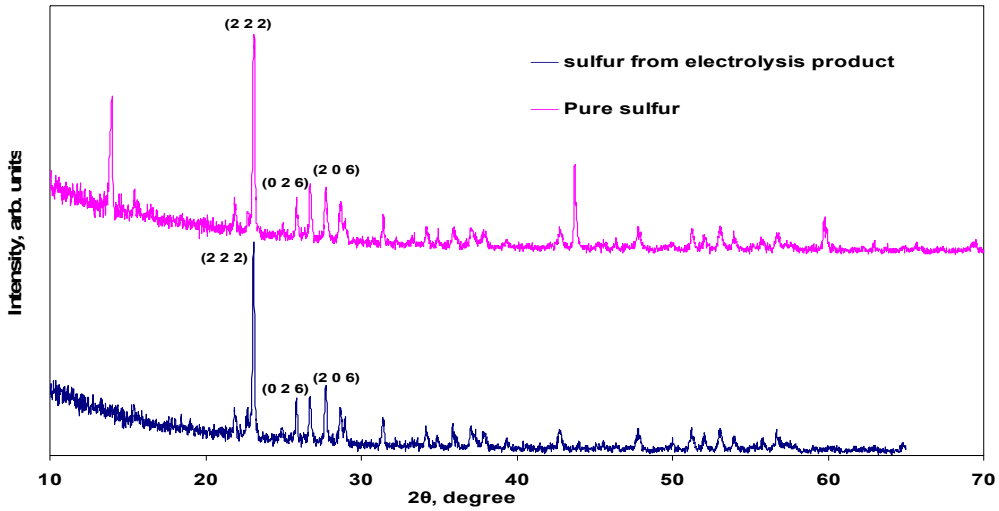

Fig.2.4.2a XRD comparison of pure sulfur and sulfur from electrolysis product. The highest intensities are used in the comparison test.

XRD analysis indicates the molten sulfur produced freezes to monoclinic crystals on cooling. Figure 2.4.2a is an XRD pattern observed on the sulfur produced and that from pure sulfur as received. This pattern matches a reference library pattern of monoclinic sulfur.

DSC analysis indicates the molten sulfur produced is the monoclinic crystal when compared to the commercial supplied sulfur. Figure 2.4.2b compares the melting point of sulfur from the electrolysis product to that of pure sulfur as received. They both have the same melting point at 119 ^{O}C, but in the electrolysis other allotropes of sulfur compound are formed such as orthorhombic sulfur which has a lower melting point than the monoclinic sulfur.

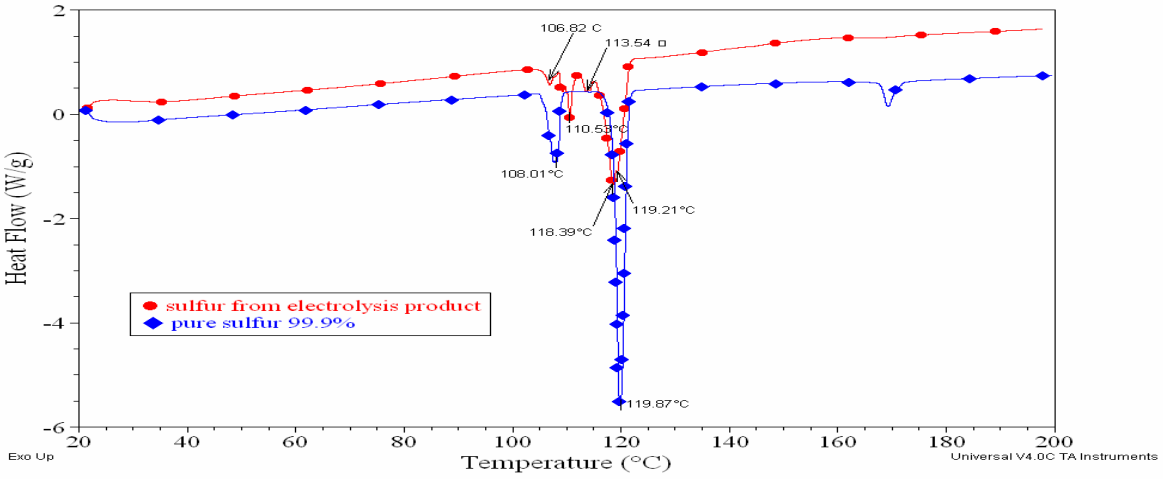

Fig.2.4.2b DSC comparison of melting points of pure sulfur and sulfur from electrolysis

Figures 2.4.2 (c) and (d) shows the SEM-EDS of the electrolyzed pellet. Figure 2.4.2(c) shows layers of yellowish sulfur deposit on the surface of the electrolyzed pellet (the area is depicted by the white background. Figure 2.4.2(d) is the energy dispersive spectroscopy (EDS) showing the wt% of sulfur deposited on top of the electrolyzed pellet.

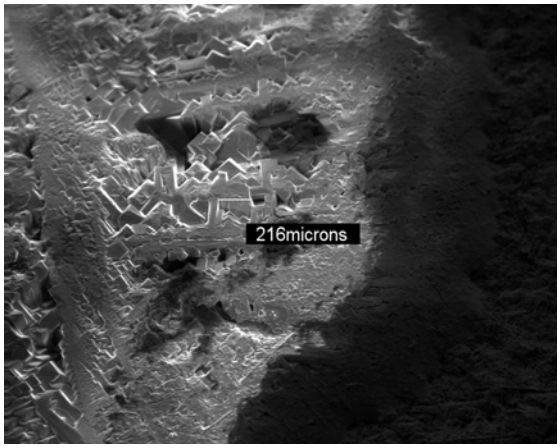

Fig. 2.4.2c: SEM image (25.0kV x40 500pm) of electrolyzed pellet showing some layers of sulfur deposit (area in white background). The dark surrounding is the background of carbon mesh used in covering the surface of the pellet before the electrolysis.

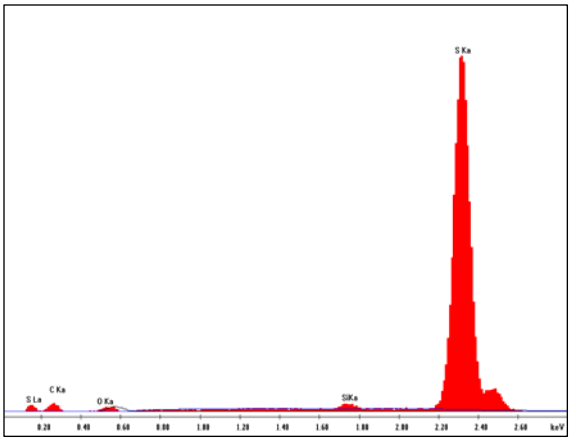

Fig. 2.4.2d: EDS spectrograph (216 microns area) showing the sulfur area formed. Sulfur formed this region is 65 wt %

Hydrogen gas produced was collected and measured by displacement of aqueous solution and identified by gas chromatography.

2.4.3. Material Balance

Hydrogen sulfide was electrolyzed for 5 hours at a steady 0.9V which produced a sustained current of 5.31 mA.

2.4.3.1. Anode

1) The half equation for the anode electrolysis is:

$$H_2S \longrightarrow 2H^+ + 2e^- + (1/8)\, S_8 \qquad (1)$$

2 moles of electrons (2 Faradays) are required to produce 1/8 mole of S_8 and 2 moles of hydrogen ions. A current of 5.31 mA for 5 hours represents 95.58 coulombs or (95.58/96,500) 0.001 Faraday. This is sufficient to produce .001 moles (.001g) of hydrogen ions and 6.25E-5 moles (.016g) of S_8.

2.4.3.2. Cathode

The half equation for the cathode electrolysis is:

$$2H^+ + 2e^- \rightarrow H_2 \qquad (2)$$

The 2 moles of hydrogen ions produced in Equation 1 are converted to 1 mole of hydrogen gas in equation 2. The 0.001 moles (.001g) of hydrogen ions produced at the anode by .001 Faraday are converted to 0.0005 moles (0.001g) of hydrogen gas at the cathode.

2.4.3.3. Gas Measurement

The gas issuing from the cathode compartment was collected and measured by displacement of water in a 50 ml burette at 25C and 102.4kPa. The observed volume was 10 ml.

Gas collected by displacement of water is saturated with water vapor whose vapor pressure at 25C is 3.17 kPa. The partial pressure of hydrogen in the burette was then (102.4 -3.17) 99.2kPa.

By the ideal gas law, .0004 moles of gas has a volume of 10 ml at a partial pressure of 99.2 kPa and temperature of 25C. Thus the measured volume of hydrogen gas is about 80% of the theoretical limit for the electrolysis as calculated in the previous section.

2.4.4. Pellet Permeability

We ran permeability tests using methanol as a surrogate for H_2S. These molecules are both water soluble and have similar molecular weights but methanol is much less toxic. Studies of methanol permeability permit a direct comparison between $CsHSO_4$ and Nafion for which previous studies have shown high methanol permeability rates[11]. Also, methanol might provide a more severe test since the lower solubility of H_2S and the large size of the sulfur atom is likely to make the H_2S diffuse more slowly.

A fortuitous finding was that treatment with methanol vapor dramatically improves the impermeability of the $CsHSO_4$ pellets. Pellets that have been run under high temperature methanol exhibit impermeability to methanol and methane. This contrasts with Nafion through which methanol passes rapidly.

Ultimately, the impermeability of the $CsHSO_4$ will have to be verified in experiments with H_2S but the amount of handling of this more dangerous gas will be reduced by first establishing procedures and parameters with the more benign methanol.

2.5. CONCLUSIONS

A system for materials handling and electrolysis has been fabricated and successfully applied to production of hydrogen and sulfur by electrolytic decomposition of hydrogen sulfide using a solid $CsHSO_4$ electrolyte. The proof of principal for this process is now accomplished.

An experiment using an anode catalyst composed of ruthenium (IV) oxide/p-Dichlorobenzene/cesium hydrogen sulfate/platinum black and cathode catalyst composed of platinum black/p-Dichlorobenzene has given the best result so far and will be used as a benchmark for further development.

Analysis of a commercial size model has indicated that electrochemical dissociation of hydrogen sulfide waste streams in IGCC power plants can be economically lucrative. This is potentially such a cost effective procedure it can have a positive influence on the cost effectiveness of IGCC energy.

Treatment with methanol vapor has produced an improvement in the impermeability of $CsHSO_4$ membranes to both methanol and methane.

GRAPHICAL MATERIALS LIST

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LIST OF ACRONYMS AND ABBREVIATIONS

PC=Pressed on Carbon
Y=Used
N=Not used
RuO_2=Ruthenium dioxide
WC=Tungsten Carbide
Pt.b=Platinum black
V_2O_5=Vanadium pentoxide
Cat=Catalyst
m A= Milliamperes
kPa= kilo Pascal
atm= Atmospheric
psi= Pounds per square inch
R = ideal Gas constant, J/mol K
C = amount of electrical charge, Amp/sec
F = Faraday's constant, C/equiv
XRD = X-Ray Diffraction
DSC = Differential Scanning Calorimetry
SEM = Scanning Electron Microscopy
MEA = Membrane Electrode Assemblies
EDS = Energy Dispersive X-Ray Spectroscopy